

<p>92-410421/50 A25 DAIL 91.04.04 DAICEL CHEM IND LTD *JP 04306228-A 91.04.04 91JP-071882 (92.10.29) C08G 65/20 Prepn. of poly(oxytetramethylene)glycol efficiently by polymerising tetrahydrofuran in presence of solid catalysts of composite metal oxide and carboxylic acid anhydride, used for e.g. engineering plastic C92-182149</p> <p>Prepn. of poly(oxytetramethylene)glycol comprises polymerising tetrahydrofuran in presence of both: (a) solid catalysts composed of composite metal oxides of the formula (1), and (b) carboxylic acid anhydrides.</p> <p style="text-align: center;">M_xO_y (1)</p> <p>M = metal; and x and y = 1, 2 or 3.</p> <p><u>ADVANTAGES/USES</u> The solid catalyst is easily recovered from the reaction mixt. and reactivated. The poly(oxytetramethylene)glycol (POTG) is obtd. easily and is used as a raw material for polyurethanes, polyetheresters, polyether-</p>	<p>A(2-A6, 2-A7, 5-H5, 10-D3)</p> <p>(ester)amides, surfactants, or engineering plastics or for medical uses.</p> <p><u>CATALYSTS</u> The catalysts pref. comprise metal oxides such as Al_2O_3, SiO_2, TiO_2, ZrO_2, WO_3 or ZnO_2. Pref. catalysts include $Al_2O_3-SiO_2$, SiO_2-TiO_2, SiO_2-ZrO_2 and TiO_2-ZrO_2. The catalyst is prepd. as follows; (1) metal compds. e.g. metal alkoxides, metal chlorides, metal oxychlorides are added to ammonia to ppt. composite metal oxides, and (2) the pptes. are crushed to particles (200-500 mesh pass) and calcined at 300-600°C.</p> <p><u>EXAMPLE</u> Silica-alumina solid catalyst (alumina content = 70%) obtd. by calcining at 500°C for 4 hrs. in an air atmos. is packed in a piston-flow type reactor (dia. = 30mm; length = 200mm). 20ml/hr. of THF contg. 3.8 wt. % of acetic anhydride is fed at 40°C and polymsn. is carried out for 132 hrs. continuously to give 404.3g of poly(oxytetramethylene)glycol diacetate ($M_n = 1,020$).</p> <p style="text-align: right;">JO4306228-A+</p>
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<p>The catalyst is removed from the reactor and washed with THF. After drying, the catalyst is charged to a tube (dia. = 30mm, length = 400mm) and calcined at 500°C for 7 hrs. under air.</p> <p>THF is polymerised by the same way in presence of the reactivated catalyst to give 406.2g of poly(oxytetramethylene)glycol diacetate ($M_n = 1,060$). (5ppW156DwgNo0/0).</p>	<p style="text-align: right;">JO4306228-A</p>
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